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Final Report
on
Low Temperature Viscosity
and
Density of Methanol-Water Mixtures

Contract No. NAS8-21378

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Abstract

This is the final report on the measurement of low temperature viscosity and density of three different methanol-water solutions. The viscosities and densities are reported for mixtures containing 83, 80, and 77 Wt. percent methanol over a temperature range of 10° C. to -100° C.

The experimental data and descriptions of the experimental equipment and procedures are given.

Experimental Data

This section contains the composition, viscosity, and the density of the three methanol-water mixtures studied.

			1	1	l		
Phase	I		<u>I</u>	<u>II</u>		<u>III</u>	
Density @ 20° C.,Gm/ml	0.8471		0.8453		0.8392		
Wt.% CH ₃ OH	79.9		77.0		83.0		
42 .	Viscosity	Density	Viscosity	Density	Viscosity	Density	
Temp., OK	Centistoke	Gm/ml	Centistoke	Gm/ml	Centistoke	Gm/ml	
TO 283.16	1.64	0.8553	1.75	0.8623	1.52	0.8466	
32 273.16	2.07						
< 258.16	3.06	0.8759	3.38	0.8821	2.75	0.8671	
- 4 253.16	3.53	•	ALTERNATION AND AND AND AND AND AND AND AND AND AN		ALCON THE PROPERTY OF THE PROP		
233.16	6.87	0.8954	7,93	0.9014	5.87	0.8874	
- 7 213.16	16.4			•	en e		
- 98 208.16	21.3	0.9155	26.5	0.9211	16.9	0.9080	
-//2. 193.16	55.0	•					
100 183.16	127.	0.9364	176.	0.9411	. 86.8	0.9295	
147 173.16	359.	0.9456	538.	0.9500	229.	0.9390	

Experimental Procedure and Equipment

Preparation of Solutions

The mixtures were made from Analytical Grade, Anhydrous, Acetone-free, Methanol manufactured by Mallinckrodt Chemical Works, and distilled water. The specified minimum purity of the methanol was 99.5%. After the mixtures were prepared by weighing the methanol and distilled water, the density of each mixture was determined by pycnometer at temperatures near 20° C. These densities were then corrected to 20° C.

Viscosity Measurement

The falling cylinder viscometer was used to determine some of the viscosity of the 80% methanol mixture. The description of this equipment is given in Exhibit A.

The major portion of the viscosity measurements were made with a calibrated Cannon-Ubbelhode Viscometer. The data obtained with this viscometer agreed closely with those obtained with the falling cylinder instrument. This viscometer is shown in Exhibit B.

Density Measurement

The densities of the mixtures were determined by measuring the volume occupied by a weighed amount of mixture contained in a calibrated glass cell. Exhibit C is a diagram of the cell. The cell was filled to a certain level with a known weight of the mixture and placed in the constant temperature bath. At each temperature at which the viscosity was measured, a cathetometer was used to measure the level of the same mixture in the stem of the cell. The density was then computed from the weight and the volume of the mixture. The density determinations are considered to be accurate to 0.1%.

Constant Temperature Bath

The constant temperature bath consisted of an agitated glass Dewar flask with two unsilvered strips to allow visual observation. An 80% methanol-water mixture was used as the bath fluid. The bath was cooled by vaporizing liquid nitrogen in a cooling coil within the bath, and the temperature was controlled by the addition of electrical heat through a nichrome wire coil.

The temperature control system consisted of a platinum resistance thermometer, a Mueller Bridge, a D-C Null Detector, a D-C Amplifier, and the nichrome wire heating coil. The resistance corresponding to the desired temperature was set on the Mueller Bridge. The difference between the bridge setting and the resistance of the thermometer represented the deviation in the temperature from the desired value. This error signal was sent to the Null Detector which amplified the signal and provided the input for the D-C amplifier. The D-C amplifier then amplified the error signal to provide the electrical power to the nichrome heater. This system permitted the measurement and control of the temperature to within 0.01° C.

EXHIBIT A

Viscosities of Methane and Propane at Low Temperatures and High Pressures

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The viscosities of methane and propane were determined at pressures to 5,000 lb./sq. in. abs. over a temperature range from -170° to 0°C . and -100° to 0°C ., respectively, and the densities of propane were measured at pressures to 5,000 lb./sq. in. abs. over the temperature range from -100° to 0°C. Reproducibility of the viscosity data was about ± 1.2% and for the most part agreement with literature values was within ± 2%. The estimated accuracy of the density measprement was ± 0.5%. The data reported were well correlated in terms of residual viscosity vs.

Although the viscosity of methane in the superambient temperature region has been investigated extensively (1 to 3, 5, 8, 9, 11, 19, 21), there are relatively few data reported at low temperatures. Pavlovich and Timrot (18) reported the viscosity of methane at temperatures to -161°C. and pressures to 2,940 lb./sq.in.abs. Ross and Brown (19) measured the viscosity of methane at temperatures to -50°C. and pressures to 10,000 lb./sq.in. gauge. Barua et al. (2) made measurements on methane viscosity at temperatures to -50°C. and at pressures to 2,500 lb./sq.in. gauge. Swift et al. (28) extended the liquid viscosity range for methane from the atmospheric pressure range reported by Rossini et al. (20) to the

The data coverage for propane is similar to that of methane. At superambient temperatures a number of investigators have reported propane viscosities (1, 3, 6, 9, 11, 22, 25 to 28) while only the data of Swift (27), which extend to -65° C. and those of Rossini et al. (20), which extend from -40°C, to the freezing point, are availab at subambient temperature.

In this study, the data for methane and propane in the low temperature range have been extended to pressures of 5,000 lb./sq.in.abs. The temperature range covered for was -170° to 0°C. and for propane, -100° to these data, together with those cited above, give bly complete coverage of the viscosity-pressureire domain for methane and propane in the 208, and dense fluid regions.

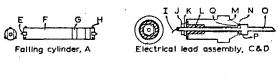
APPARATUS AND MATERIALS

The opparatus used in this investigation is similar to that reporte by Swift et al. (28) but incorporates certain modifications which extend the upper operating pressure limit to 12,000 lb./sq.in.abs. To date the apparatus has not been operated at pressures above 5,000 lb./sq.in.abs. because of the limitations of pressure measuring equipment available.

The viscometer used is of the falling cylinder type (see

Figure 1). An assembly consisting of a right circular cylinder of aluminum (A) with protrusions (E) for making electrical contact and pins (H) for aligning the cylinder concentrically

within the vertically positioned precision bore tube (B) made of AISI 316 stainless steel is used to make the viscosity measurements. Cylinder (A) is raised to the top of tube (B) by the action of a magnet [mounted externally to tube (B)] on the two mild steel rods (G) imbedded in cylinder (A). As upper protrusion (E) on cylinder (A) comes into contact with electrical lead (O) of the top electrical lead assembly (C), cylinder (A) is disengaged from the magnetic field and falls through the fluid confined in tube (B). The breaking of the electrical contact between upper protrusion (E) on cylinder (A) and electrical lead (O) of the top electrical lead assembly (C)initiates a time count on a time interval meter. The making of the electrical contact between lower protrusion (E) on cylinder (A) and electrical lead (O) of the bottom electrical lead assembly (D) terminates the time count. The tube (B) forms the ground side of the circuit in both initiation and termination steps of the time count. The electrical leads (O) are insulated from tube (B) as shown in Figure 1. Electrical lead (O) makes a nonconducting, unsupported-area pressure seal with shoulder (Q) of plug (M) via Mylar gasket (N). Insulator (L) spaces electrical lead (O) through the port in plug (M) to prevent shortcircuiting. Washer (K) and lock nut (I) hold the assembly together and are tightened to effect the initial (zero pressure) seal between electrical lead (O) and plug (M).





F Aluminum body

- A Falling cylinder Precision bore ss tube Top elec, lead assembly
- G Mild steel rod H Alignment pin D Bottom elec lead assembly E as protrusion I Electrical wire
- K Steel washer L Non-metallic insulator M ss plug N Mylar gasket O ss electrical lead

Fig. 1. Diagram of viscometer assembly.

- 65 +

The viscometer is housed in a controlled temperature bath which is a modified version of the one described by Sinor (24). The heat transfer medium for the bath is gaseous nitrogen at a pressure slightly above atmospheric pressure. The nitrogen is circulated by a high-speed centrifugal blower powered by an air motor. Liquid nitrogen at atmospheric pressure is used as coolant; the vaporized nitrogen is injected directly into the bath as heat transfer medium makeup, Coolant control is effected by controlling the back-pressure on the bath. An electrical resistance wire heater provides the amount of heat necessary to offset the liquid nitrogen vaporization load at a preset temperature level. The resistance setting on the bridge of a platinum resistance thermometer-Mueller bridged.c. null detector system sets the temperature level in the bath. A signal from the d.c. null detector reflecting the difference between thermometer temperature and set-point temperature is fed to a solid state temperature control device which in turn proportionally controls the voltage drop across the resistance wire heater.

Temperature measurement and control at all temperature levels employed in this investigation were within $\pm 0.01\,^{\circ}$ C. The placinum resistance thermometer used for this purpose was calibrated against a similar instrument, which in turn had been calibrated directly by the National Bureau of Standards. Temperature differences between thermometer and various points on the viscometer were less than 0.1 $^{\circ}$ C. as measured by thermocouples whose reference junctions were mounted on the sensing element of the thermometer.

Pressure measurements were made with a 5,000 lb./sq.in.abs. Bourdon tube gauge which had a maximum uncertainty of \pm 5 lb./sq.in. The pressure gauge was calibrated against a pressure balance accurate to \pm 1 lb./sq.in. Time of fall data was reproducible to \pm 0.2% at 98% confidence and was measured with a Beckman Model 5230R-4 time interval meter.

The density measurements reported in this investigation wew etermined with a precision bore, high-pressure burette which displaced measured volumes of fluid into the viscometer at a constant temperature and pressure. The data of Kvalnes and Gaddy (14) and of Sage and Lacey (23) were used to convert the volumes of fluid transferred to masses of fluid transferred. The accuracy of the density data reported in this study is estimated to be $\pm 0.5\%$.

Phillips pure grade (99 mole % minimum) methane and instrument grade (99.5 mole % minimum) propane were used in this investigation. These materials were introduced into the apparatus via tubes filled with ascarite and phosphorus pentoxide to remove trace quantities of water and carbon dioxide. Analyses of the two materials by F & M Model 720 chromatograph with a silica gel column are as follows:

		Ŋ	Aole %	n	
			Pro-	Bu-	
•	Methane	Ethane	pane	tane	Nitrogen
Pure grade methane	99.31	0.15	0.01		0.53
Instr. grade propane	trace		99.97	0.03	trace

CALIBRATION OF THE VISCOMETER

The theory of the falling cylinder viscometer and the method for correlating experimental calibration data in terms of the theory have been discussed in detail (15, 16). The calibration data for the viscometer used in this investigation are expressed in the form

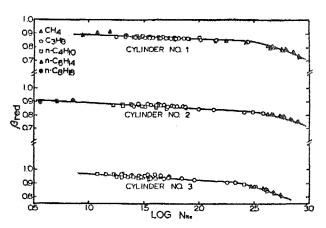


Fig. 2. Viscometer calibration plot.

$$\beta_{\rm red} = \beta_{\rm exp}/\beta_{\rm calc} = f(N_{Re}) = f(D_e s_P/\theta_\mu)$$
 (1)

where β_{exp} , the viscometer constant determined experimentally by measuring times of fall for calibrating fluids, whose densities and viscosities are known, at various temperatures and pressures, is defined as

$$\beta_{\rm exp} = \mu s / [(\sigma - \rho)\theta] \tag{2}$$

 β_{calc} , the viscometer constant derived theoretically in terms of the physical dimensions of the instrument, is defined as

$$\beta_{\text{calc}} = gD_e^2 (1 - \kappa^2)^4 (1 - \kappa)^2 / \{32\kappa^6 (1 + \kappa^2) - (1 + \kappa^2) \ln(1/\kappa) - (1 - \kappa^2)\}$$
 (3)

and D_e , the equivalent diameter for the annular gap between the falling cylinder and the viscometer fall tube, follows from the definitions for the Reynolds number N_{Re} , the friction factor f [see Equation (6.1-1) of Bird et al. (4)], and $f = 16/N_{Re}$, and is expressed as

$$D_e = 2\kappa^4 D_t [(1 + \kappa^2) \ln(1/\kappa) - (1 - \kappa^2)]/[(1 - \kappa)(1 - \kappa^2)^2]$$
 (4)

The physical dimensions of the viscometer, s, D_c , D_t and $\kappa = D_c/D_t$, are expressed as functions of temperature and pressure (16) in the relationships above so that all calibration data, expressed in terms of Equation (1), are independent of temperature and pressure effects; that is, $\beta_{\rm red}$ is a function only of the flow regime prevailing in the viscometer. $\beta_{\rm red}$ can approach unity in the limit as s/θ approaches zero and is less than unity for finite values of s/θ because entrance and exit effects, alignment pin friction, etc., are not taken into effect in the theoretical development of $\beta_{\rm calc}$. The effect of body acceleration to terminal velocity in the viscometer has been shown to be negligible at all Reynolds numbers encountered in this study (13).

The fluids for calibration were selected so as to encompass the range of Reynolds numbers anticipated in

TABLE 1. CALIBRATION RESULTS

Cyl. No.	Cyl. dia., in.	$\log(N_{Re})tr$	A_0	A_1	% Error*	\mathcal{B}_0	<i>B</i> 1	B_{v}	% Error†	log(NRe)maxt
1 2	0.3077	2.446	0.9351	-0.03870	±0.61	0.3633	0.5441	-0.1427	±0.81	2.917
	0.3078	2.488	0.9353	-0.04462	±0.85	0.6431	1.2495	-0.2656	±0.83	2.904
	0.3087	2.327	1.0207	-0.04888	±0.81	0.3075	1.1564	-0.2730	±0.65	2.750

[†] Percentage errors for Equations (5) and (6), respectively, are reported as average percentage errors of the data points from the regression curves. 1 $\log(N_{Re})_{max}$ is the largest $\log(N_{Re})$ encountered during the calibration.

The diameter of the viscometer tube was 0.3140 in. All dimensions of the apparatus were measured at 25°C.

the experimental investigation and to insure internal consistency of viscosity data insofar as possible. The fluids used (all Phillips 99 mole % minimum purity hydrocarbons) and the literature references for viscosity and density data were:

Fluid	Viscosity literature reference	Density literature reference
Methane	2	14, 29
Propane	26	23
n-Butane	10	23
n-Hexane and n-Octane	20	20

Three falling cylinders, each falling in the same viscometer tube, were calibrated, giving the results shown in Figure 2 and Table 1. The form of the functional relationship of Equation (1) was determined for each falling cylinder by regression analysis after observing that the data depicted in Figure 2 indicated transition points such that the form for regression at Reynolds numbers less than the transition value should be

$$\beta_{\text{red}} = A_0 + A_1 \log(N_{Re}) \tag{5}$$

while at Reynolds numbers greater than the transition value, the 1 gression form should be

$$\beta_{\text{red}} = B_0 + B_1 \log(N_{Re}) + B_2 [\log(N_{Re})]^2$$
 (6)

To determine experimental viscosities, the calibration data are used as follows:

- 1. β_{calc} , s, and D_e are computed for the temperature and pressure at which the experiment was conducted.
- 2. A trial value of μ is assumed and the Reynolds number is computed by using the experimentally determined

Time 2. Recommended Viscosity Values for Liquid, Vapor, and Fluid Methane

Pressure,	Temperature, °C.							
lb./sq. in. abs.	0.	-25°	—50°	60°	—70°	-80°		
			Viscosity,	micropoi	se			
14.7	101*	93*	85°	83	80	76		
600	111*	103°	96°	92	90	94		
800	115	108*	104*	103	113	235		
1,000	120	116	118	127	185	288		
1,500	138	144	184	229	280	345		
2,000	161	182	244	287	333	391		
3,000	214	256	330	364	403	453		
4,000	263	314	386	420	456	508		
5,000	305	361	432	463	501	555		
	-100°	—120°	—140°‡	-150°	—160°	—170°		
14.7	71	64	58	55	52	1,440		
Dew point!	75	66	58	55	52	50		
Bubble								
point	385	542	767	940	1,140	1,440		
enn	405	570	810	990	1,190	1,510		
1,000	438	599	840	1,020	1,230	1,560		
1,500	477	631	878	1,070	1,280	1,620		
2,000	512	664	917	1,110	1,330	1,680		
3,000	573	727	990	1,200	1,420	1,810		
4,000	627	788	1,060	1,290	1,520	1,930		
5,000	676	847	1,140	1,370	1,620	2,060		

^{*} Values obtained from the data of Barna et al. (2).

† Dew point end bubble point pressures are from the literature (17).

† The vapor phase viscosities at temperatures below -140°C, were extrapolated from the higher temperature data.

TABLE 3. RECOMMENDED VISCOSITY AND DENSITY VALUES FOR LIQUID PROPANE

Pressure, lb./sq. in. abs.	0° Viscosity density Micropoise g./ec.		der	20° osity osity opoise	—40* Viscosity density Micropoiseg./cc.	
Bubble point* 1,000 2,000 3,000 4,000 5,000	1,260 1,380 1,490 1,600 1,710 1,810	0.528 0.540 0.551 0.561 0.569 0.576	1,550 1,670 1,800 1,910 2,030 2,150	0.554 0.564 0.573 0.582 0.589 0.595	1,940 2,070 2,210 2,340 2,470 2,600	0.578 0.587 0.594 0.602 0.608 0.613
1,000 2,000 3,000 4,000 5,000	2.450 2,600 2,750 2,910 3,060 3,220	0.602 0.608 0.616 0.622 0.627 0.632	3,160 3,320 3,500 3,680 3,880 4,060	0.624 0.630 0.636 0.641 0.645 0.651	4,290 4,520 4,760 5,020 5,270 5,540	0.646 0.651 0.655 0.660 0.663 0.669

[•] Bubble point pressure values are from the literature (12).

fall time and the density at the temperature and pressure of the experiment (either from literature or experiment).

- 3. The appropriate regression relationship [Equation (5) or (6) for the falling cylinder used] is selected for the Reynolds number computed in step 2.
- 4. $\beta_{\rm exp}$ is computed from $\beta_{\rm calc}$, $N_{\rm Re}$, and the appropriate regression relationship.
- 5. μ is computed from Equation (2) by using β_{exp} , s, σ , and experimental data for θ and ρ .
- 6. This iterative procedure is continued until the assumed and computed values of μ agree to within an assumed the sum of the sum

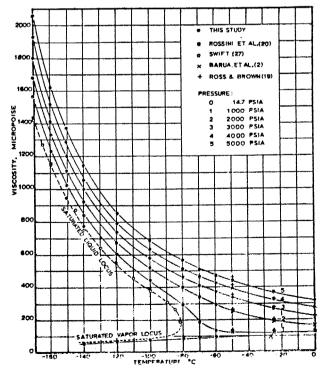


Fig. 3. Methane viscosity vs. temperature.

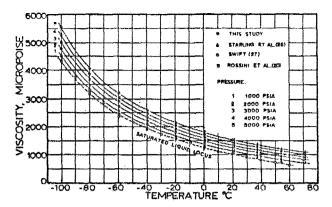


Fig. 4. Propone viscosity vs. temperature.

signed tolerance. This procedure is facilitated by using a digital computer.

In the experimental investigation, methane at most and propane at all of the temperatures and pressures covered were in the liquid or dense fluid states such that the Reynolds numbers were below the transition value. Certain of the low pressure-methane viscosity data were obtained at Reynolds numbers above the transition value. However, the highest log (N_{Re}) value encountered in this case was 2.992, so that only a modest extrapolation of the calibration was required.

EXPERIMENTAL RESULTS AND DISCUSSION

Experimental viscosity data were determined for methane at pressures to 5,000 lb./sq.in.abs. over the temperature range from -170° to 0° C. and experimental viscosity and density data were determined for propane at pressures to 5,000 lb./sq.in.abs. over the temperature range from -100° to 0° C. The viscosities for methane and propane were determined over the complete temperature and pressure ranges by using cylinder No. 1, while data were taken by using cylinders No. 2 and 3 for selected isotherms within the respective temperature ranges for methane and propane. The precision of the experimental viscosity measurements, determined by comparing the results obtained from the three cylinders for methane and propane, was \pm 1.2% (13).

Table 2 presents the recommended values of viscosity for methane and Table 3 presents the recommended values of viscosity and density for propane. The viscosity data used to prepare these tables are those obtained from cylinder No. 1. For methane, the tabular information was extended to low pressures by using the viscosity data reported by Barua et al. (2) at 0°, -25°, and -50°C. The tabulated information presented was obtained from smoothed curves of viscosity (or density) as a function of pressure with temperature as a parameter. The recommended values presented in Tables 2 and 3 agree with the data of cylinder No. 1 to within \pm 1%. The bubble and dew point viscosity values were obtained by extrapolation to the saturation pressures reported in the literature (12, 17). Vapor viscosities of methane at temperatures below -140 °C. were obtained by extrapolation of higher temperature data, since no experimental vapor data were available at these temperatures.

The experimental data for methane and propose from cylinder No. 1 are presented in Figures 3 and 4, respectively, as graphs of viscosity vs. temperature with pressure as a parameter. These figures also compare the results of this study with those reported in the literature. For methane, the low-pressure liquid viscosities of this study agree with those reported by Rossini et al. (20) to

within ± 1.4% but are consistently higher than those reported by Swift (27) by about 2.5%. Although Pavlovich and Timrot (18) reported liquid viscosities for methane. their data do not appear to be reliable since they are considerably higher than the values determined in this investigation and those reported by Rossini et al. and Swift (about 40% higher at -161°C.). In the higher temperature region for methane, the results of this study agree well with those of Barua et al. (2) which is to be expected, since those data were used in the calibration of the viscometer. The data reported by Ross and Brown (19) were consistently higher than those of this investigation by as much as 8.3%. The data obtained in the present study extrapolate smoothly to the superambient temperature values reported by Baron et al. (1), Carmichael et al. (5), and Giddings (11). For propane, the low-pressure liquid viscosities of this study agree with those reported by Swift (27) and Rossini et al. (20) to within ± 2.5% on the average, although the data of Swift were consistently lower by about 1.5%, and those of Rossini et al. were consistently higher at temperatures approaching the normal boiling point of propane by as much as 5.7%. Again, the data of this study extrapolate smoothly to the superambient temperature data reported by Starling et al. (26) which is to be expected, since these data were used in the viscometer calibration.

Figures 5 and 6 present the residual viscosity of methane and propane, respectively, as a function of density and compare the results of this study to those of other investigators. For propane, the atmospheric pressure viscosities used in computing residual viscosity were obtained by extrapolation of literature values (6, 26). The average deviation of methane data of this study from the curve presented in Figure 5 for densities greater than

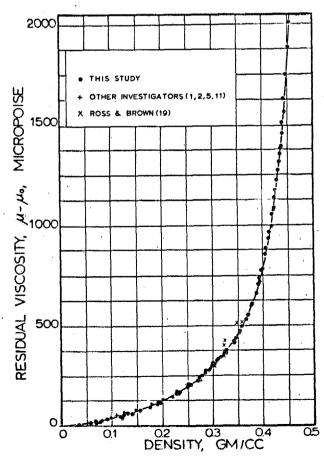


Fig. 5. Methane residual viscosity vs. density.

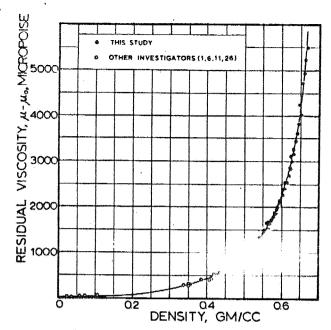


Fig. 6. Propane residual viscosity vs. density.

0.25 g./cc. is ± 1.0%, while for propane the average deviation from the curve presented in Figure 6 for all densities investigated is \pm 1.5%.

CONCLUSIONS

A falling cylinder viscometer is described which is capable of measuring viscosities of light hydrocarbons in the gas, liquid, and dense fluid regions at pressures to 12,000 lb./sq.in.abs. over the temperature range from -170° to 100°C., while fluid densities are measured concurrently in the same instrument.

The viscosities of methane and propane were determined at pressures to 5,000 lb./sq.in.abs. over a temperature range from -170° to 0°C. and -100° to 0°C., respectively. The reproducibility of these data was on the average ± 1.2%, and agreement with literature values was for the most part within ± 2.0%. Densities of propane were determined at pressures to 5,000 lb./sq.in.abs. over the temperature range from -100° to 0°C. with an estimated accuracy of $\pm 0.5\%$.

The data of this study were used to extend the residual viscosity-density correlation well into the high density region. For methane, a smooth curve of the residual viscosity-density relationship represented the data of this study at densities greater than 0.25 g./cc. to within \pm 1% on the average and for propane at all densities studied, the average deviation from the smooth curve was $\pm 1.5\%$.

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NOTATION

 A_0 , A_1 = coefficients of Equation (5), dimensionless B_0 , B_1 , B_2 = coefficients of Equation (6), dimensionless = O.D. of falling cylinder, cm.

= equivalent diameter of viscometer [Equation D_{ϵ} (4)], cm.

= I.D. of viscometer fall tube, cm.

= friction factor, = $16/N_{Re}$, dimensionless local acceleration of gravity, cm./sec.2

Reynolds number [Equation (1)] for viscometer, dimensionless

distance of fall in the viscometer, cm.

Greek Letters

 β_{calc} = theoretical viscometer constant [Equation (3)],

experimental viscometer constant [Equation (2)], β_{exp} cc./sec.2

reduced viscometer constant [Equation (1)], dimensionless

time of fall, cm./sec.

= D_c/D_t , dimensionless

viscosity, g./(cm.)(sec.)

fluid density, g./cc.

falling cylinder density, g./cc.

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EXHIBIT B

Certificate of Calibration

Viscometer No. A313

CANNON-UBBELOHDE TYPE (Standard Test ASTM D 445)

Viscometer Constant

0.1063

Centistokes/Second

. The viscometer constant is the same at all temperatures.

To obtain viscosity in centistokes multiply the efflux time in seconds by the viscometer constant.

CALIBRATION DATA AT 100° F.

Viscosity Standard	Viscosity Centistokes	Efflux Time Seconds	Viscometer Constant Centistokes/Second
7 H	22.68	213.3	0.10633
71	35.58	334.9	0.10624
•	Act of the second		** # ** *

Average = 0.1063

R. E. Manning, Ph.D.

W. A. LLOYD, Ph.D.

REGISTERED PROFESSIONAL ENGINEERS

CANNON INSTRUMENT CO.

STATE COLLEGE, PENNSYLVANIA

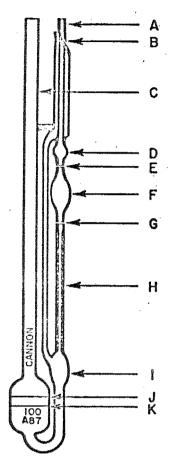
Viscosities of the standards used in calibrating were established in Master Viscometers as described in Ind. Eng. Chem. Anal. Ed. 16, 708 (1944) by M. R. Cannon. This method has been favorably checked at the National Bureau of Standards by Swindells, Hardy and Cottington and their work is published in the Journal of Research of the National Bureau of Standards, Vol. 52, No. 3 March 1954, Research Paper 2479.

Viscosities are based on the new value for water adopted by the National Bureau of Standards and The American Society for Testing Materials July 1, 1953. The new viscosity basis is 1.0038 centistokes for water at 68° F. The gravitational constant, g, is 980.1 cm/sec² at the Cannon Instrument Company. The gravitational constant varies up to 0.1% in the United States. To make this small correction in the viscometer constant, multiply the above viscometer constant by the factor [g (at your laboratory) /980.1].

See instructions for use of the Cannon-Ubbelohde Viscometer on the reverse side.

Instructions for the use of The Cannon-Ubbelhode Viscometer*

See also ASTM D 445



- 1. Clean the viscometer using suitable solvents and dry it by passing clean, dry filtered air through the instrument to remove the final traces of solvents. Periodically, traces of organic deposits should be removed with chromic acid.
- 2. If there is a possibility of lint, dust, or other solid material in the liquid sample, filter the sample through a fritted glass filter or fine mesh screen.
- 3. Charge the viscometer by pouring enough sample through tube C to fill the lower reservoir about one-half full.
- 4. Place the viscometer into the holder and insert it into the constant temperature bath. Vertically align the viscometer in the bath if a self aligning holder has not been used.
- 5. Allow approximately 20 minutes for the sample to come to bath temperature.
- 6. Place a finger over tube B and apply suction to tube A until the liquid reaches the center of bulb D. Remove suction from tube A. Remove finger from tube B, and immediately place it over tube A until the sample drops away from the lower end of the capillary into bulb I. Then remove finger and measure the efflux time.
- 7. To measure the efflux time, allow the liquid sample to flow freely down past etch mark E, measuring the time for the meniscus to pass from etch mark E to etch mark G to the nearest 0.1 second.
- 8. Calculate the viscosity of the sample by multiplying the efflux time by the viscometer constant.
- 9. Without recharging the viscometer, make check determinations by repeating steps 6 to 8.

U.S. Patent 2,805,570

Cannon-Ubbelohde Type

for Transparent Liquids

Excellent inexpensive holders are listed in our Viscometer Bulletin.

RECOMMENDED VISCOSITY RANGES FOR THE CANNON-UBBELOHDE VISCOMETERS

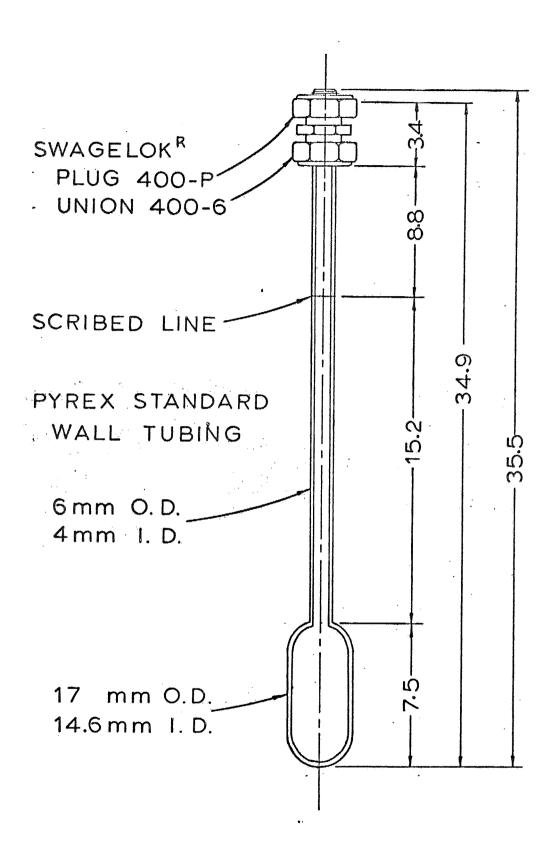
Approximate Constant ize Centistokes/Second				Range Saybolt Universal, sec	,
0.002 0.004	0.8	to	1.6 3.2	•••••••••••••••••••••••••••••••••••••••	
0.015	3	to	12	36 to 66	
0.1	20	to	80	98 to 370	
0.5	100 240	to	400 960	460 to 1900 1100 to 4400	
2.5 8	500 1600	to	2000 6400 16000	2300 to 9300 7400 to 29600 18500 to 74100	
	Centistokes/Second 0.002 0.004 0.008 0.015 0.035 0.1 0.25 0.5 1.2 2.5	Centistokes/Second C 0.002 0.3 0.004 0.8 0.008 1.6 0.015 3 0.035 7 0.1 20 0.25 50 0.5 100 1.2 240 2.5 500 8 1600	Centistokes/Second Centistokes/Second 0.002 0.3 to 0.004 0.8 to 0.008 1.6 to 0.015 3 to 0.035 7 to 0.1 20 to 0.25 50 to 0.5 100 to 1.2 240 to 2.5 500 to 8 1600 to	Centistokes/Second Centistokes 0.002 0.3 to 1.6 0.004 0.8 to 3.2 0.008 1.6 to 6.4 0.015 3 to 12 0.035 7 to 28 0.1 20 to 80 0.25 50 to 200 0.5 100 to 400 1.2 240 to 960 2.5 500 to 2000 8 1600 to 6400	Centistokes/Second Centistokes Saybolt Universal, second 0.002 0.3 to 1.6

For information for other sizes and viscometers, write to us.

-13-

EXHIBIT C

-14-



DENSITY CELL

1/2 SIZE
DIMENSIONS IN cm